**Insulating Biomaterials N01-NS-2-2347**

**Sixth Quarterly Progress Report January-March, 2004**

**National Institutes of Health**

**National Institute of Neurological Disorders and Stroke**

**Neural Prosthesis Program**


**InnerSea Technology**

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The goal of the Insulating Biomaterials work is to identify and evaluate materials, coatings, and assembly techniques suitable for protection of integrated circuit devices being considered for neural prosthetic applications.

## New Instrumentation System

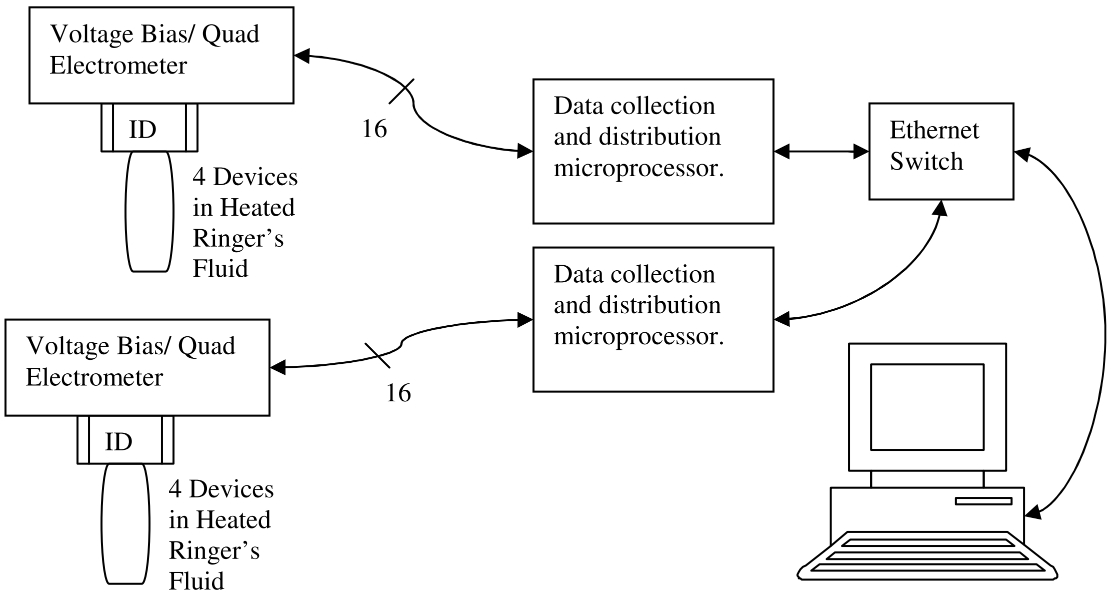



**Figure 1:** System sketch of instrumentation system. Electrometer and device ID boards have been designed and are ready for assembly and testing. Data collection and distribution microprocessor board is currently in design. A 16 channel Ethernet switch for collecting data from intermediate modules is commercially available for nominal cost.

A complete data acquisition module was designed based on results reported earlier. This module consists of a quad electrometer array, continuity testers, calibrators, device ID reader, bias voltage digital-analog converter, and bi-directional digital data communication bus all controlled by a small microprocessor. As shown in Figure 1, each module will plug into a test tube or jar equipped with a device attachment and identification module. Up to 16 modules will receive power from and communicate with an intermediate microprocessor that will assemble or distribute information to and from a central data acquisition computer. Communication between the intermediate microprocessor and the central computer will through an Ethernet interface.

When fully implemented, this approach will cost less than $200/module including support circuitry, will be virtually limitless for expansion as devices continue to survive for many years, and is virtually fault proof as each measurement sequence will verify the device code and will calibrate the instrumentation prior to each measurement.

In addition to the prototype circuit boards that were tested, an Ethernet communications module was successfully interfaced with a microcontroller. Communications between the microcontroller and the Ethernet module was accomplished at 215kpbs while communication between the Ethernet module and a remote PC computer was accomplished with standard net protocol at 100Mbps.

Next quarter the complete system will be designed and fabricated on a small scale to finalize the designs.

**Battery Testing**

Figure 2: Battery set used to evaluate cure cycles at 125$^{o}$C, 150$^{o}$C, and controls.

In order to determine the effects of the silicone cure schedule on battery lifetime, three sets of Tadiran hermetic sealed lithium thionyl chloride batteries were first heat cycled according to standard cure schedules and then assembled with standard 10k load resistors (Figure 2). Battery voltage was periodically monitored and the “end of life” for the batteries was defined as when the battery voltage fell to less than 2.5 volts. Results are displayed in Figure 2. It was somewhat surprising that the “Backup” batteries exhibited highest capacity while the “Extended” temperature range batteries exhibited lowest capacity. It was clear that the cure cycles had little effect on battery capacity.

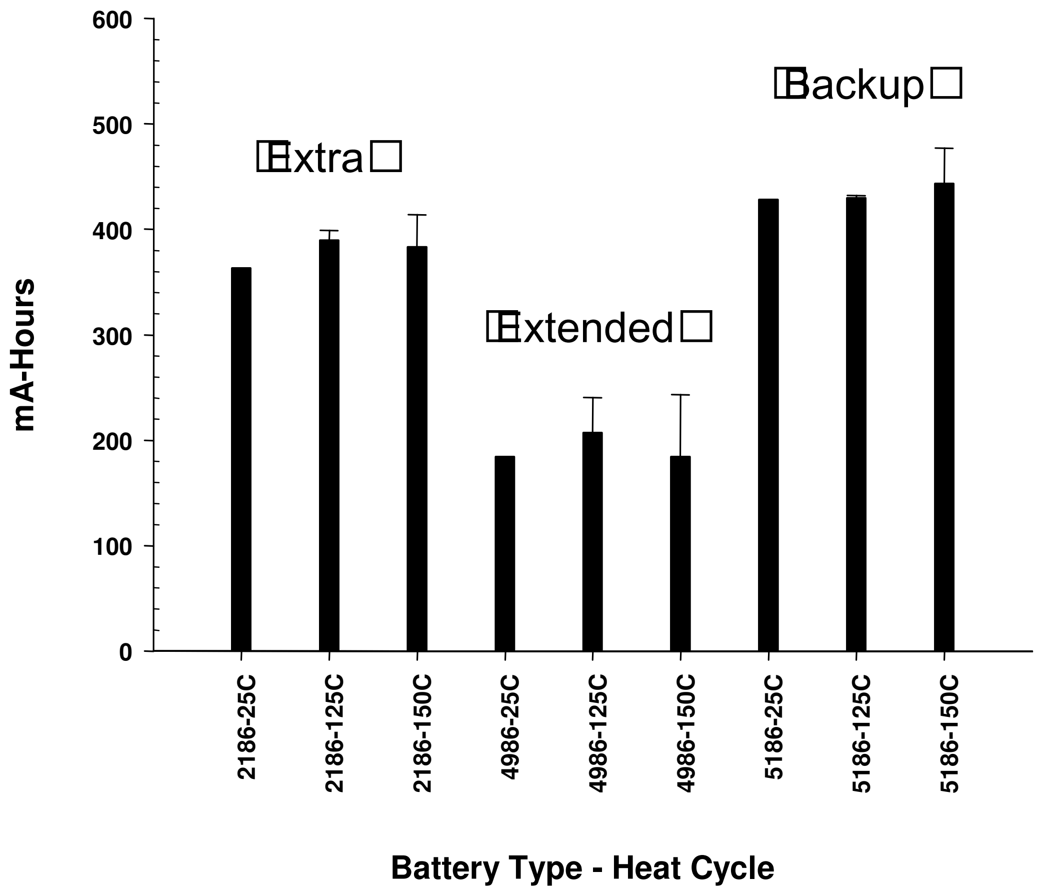



**Figure 3: Effect of heat cycle on battery performance for three types of Tadiran batteries. 10kΩ load batteries provided current drain. Milliampere-Hours computed by integrating area under discharge curve down to 2.5 volts.**

## Adhesion Promoters

A variety of adhesion promoters have been identified as possible enhancements for bonding Liquid Silicone Rubbers (LSRs) to quartz or glass substrates. The concept is that the methoxy or ethoxy groups will exchange with surface hydroxyl groups there by allowing covalent bonding of the silane. The presence of free vinyl end groups allows direct cross-linking with the typical platinum catalyzed silicone reaction shown in Figure 4. Typical "Cross-Linker' chemistry, used to increase the stiffness and tear resistance of the silicones can also directly link to available vinyl groups from adhesion promoters as shown in Figure 5. Examples of the adhesion promoters being considered are shown in Figure 6. Others are being considered. Direct testing of adhesion promoters has been delayed by variable results from the peel testing but may begin shortly. Because peel testing has been quite variable, testing of the adhesion promoters using sensitive triple track surface insulation test devices will also begin next quarter.

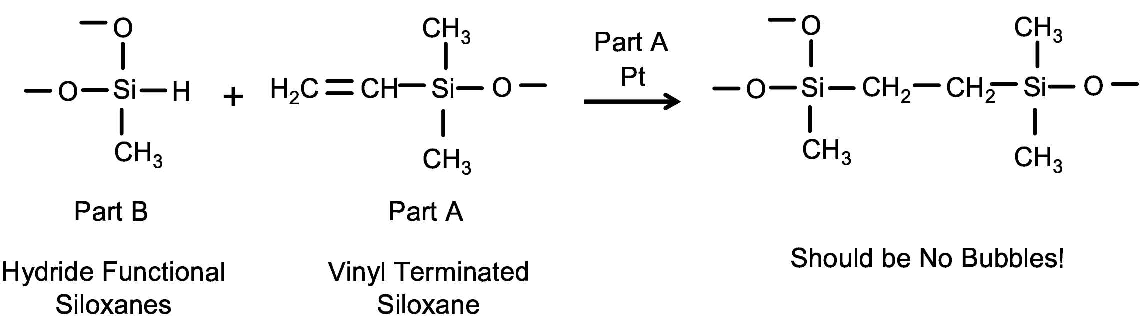



**Figure 4:** Typical 2 part platinum catalyzed silicone chemistry. Presence of free vinyl groups affords the possibility of using vinyl substituted silane coupling agents to enhance covalent bonding to silicon dioxide surfaces.

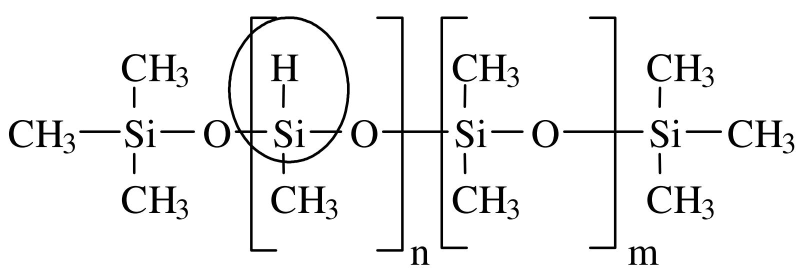
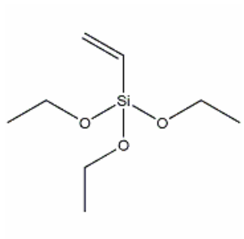
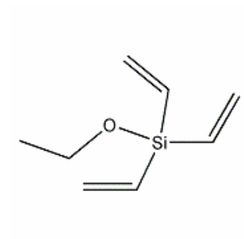


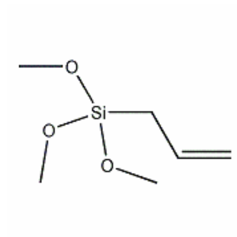
Figure 5: Cross-linker in typical 2 part platinum catalyzed silicone elastomer is reactive with multiple vinyl groups



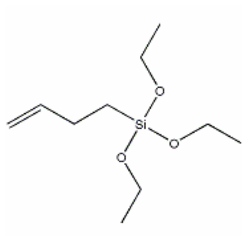
VINYLTRIETHOXYSILANE



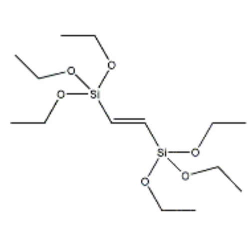
TRIVINYLETHOXYSILANE



ALLYLTRIMETHOXYSILANE



BUTENYLTRIETHOXYSILANE



BIS(TRIETHOXYSILYL)ETHYLENE

Figure 6: A variety of silane coupling agents have been identified and procured.

**Peel Testing**

Precision cut narrow Kapton tape with silicone pressure sensitive adhesive was received and all samples will now be constructed with periodic release strips to ensure that each pull begins at the silicone/substrate interface. A new formulation of silicone with very long pot life to allow vacuum removal of bubbles was also received. A variety of fiberglass tapes were also procured to determine if the fiberglass tape used for peeling the silicone in some way contributes to the variable results observed.

**LCP Substrate Testing**

Considerable effort has been underway for the past 2 years in establishing LCP as an implantable electronic circuit insulating material. A variety of electrical soak tests, mechanical tests, peel tests, and implant tests have been accomplished and will be reported on next quarter.

**CVD Synthesis of New Materials**

<u>Optimization of Organosilicon Film Deposition Conditions</u>

Work for the past few months has focused on optimization of thin film properties based on the Trivinyl-TriMethyl-Cyclotrisiloxane ($V_3D_3$) : Tertbutyl-Peroxide chemistry. Previous samples have shown good flexibility and conformation to the substrate, but were not resistive under test. In addition, samples were soluble in almost all solvents, including limited solubility in water. Optical microscopy revealed small 'bubbles' in the films, resulting from out-gassing of volatile species which had condensed/adsorbed onto the substrate during deposition. Further analysis revealed that the films were in fact losing significant thickness during post-deposition reactor blowdown, often up to 40%.

To alleviate this situation, reactor modifications were performed to allow independent control of the substrate temperature and the filament temperature. This allowed the substrate to be held at elevated temperatures (60-80$^{o}$C) without changing other process conditions. A variety of reactor conditions were examined for their effect on film properties including variations in hot filament and substrate temperatures, initiator to precursor ratio, and residence time of precursor gasses. It was observed that the elevated substrate temperature prevented adsorption of volatile species during film deposition and led to films which retained their full deposited thickness on reactor blowdown.

Physical Properties of Films

Preliminary analysis of films prepared under these new conditions has been very favorable. Films are assumed to be of very high molecular weight (~infinite molecular weight due to extensive crosslinking) and show no solubility in water, isopropyl alcohol, acetone, Tetrahydrofuran (THF), Dimethyl Sulfoxide (DMSO), or Dimethyl Acetamide (DMAC). Films are extremely adhesive to silicon substrates and show no delamination or liftoff when scored and boiled for 30min. In addition, preliminary resistance testing indicates bulk resistivity in excess of $10^{15}$ Ohm-cm. Samples of this material of varying thicknesses (1-5$\mu$m) have been prepared and provided to Innersea Technologies for long term soak testing.

Future work with this chemistry will focus in two areas. First, deposition rates for this material are extremely low (<1$\mu$m/hr). Process conditions will be optimized to increase deposition rate while retaining desirable film material properties (solubility, resistivity, etc). Preliminary results indicate that increases in deposition rate of an order of magnitude or more are possible through manipulation of reactor temperature and pressure. Once the deposition rate has been optimized, work will then focus on coating of wire samples to verify film flexibility and resistivity on these substrates.